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PATENT ABSTRACTS OF JAPAN

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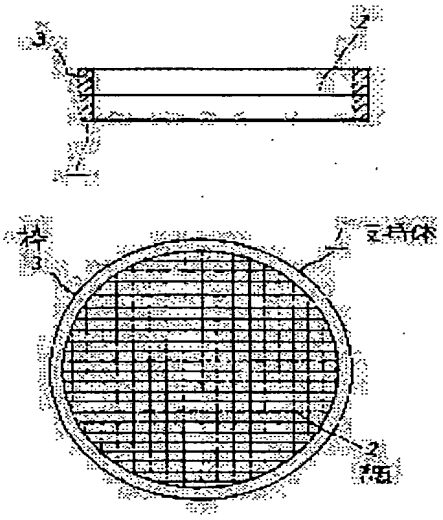
HIRAI KEIZO

(54) CHEMICAL VAPOR DEPOSITION METHOD

(57)Abstract:
PURPOSE: To enable chemical vapor deposition without leaving a support mark by placing a substrate to be subjected to the vapor deposition on the support having the net of carbon fibers coated with thermally decomposable carbon or silicon carbide.
CONSTITUTION: The net 2 of carbon fibers is fixed by holding between two frames 3 of artificial graphite, and the net 2 is coated with thermally decomposable carbon or silicon carbide to obtain a support 1. The substrate to be subjected to the vapor deposition is placed on the support 1, and the chemical vapor deposition is carried out. The substrate contacts linearly with the carbon fibers, and a reactive gas penetrates between the substrate and the support 1 because of the finely uneven surfaces of the carbon fibers, so the vapor deposition is carried out on the whole surface of the substrate without leaving the support mark.



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⑫公開特許公報（A）　昭61－124574

⑤Int.Cl.⁴　識別記号　庁内整理番号　⑬公開　昭和61年(1986)6月12日
C 23 C 16/26　8218－4K

審査請求　未請求　発明の数 1　（全3頁）

⑭発明の名称　化学蒸着法

⑳特　願　昭59－244895
㉒出　願　昭59(1984)11月20日

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明　細　書

1. 発明の名称

化学蒸着法

2. 特許請求の範囲

1. 熱分解炭素又は炭化珪素を被覆した炭素繊維の網からなる支持体の上に被蒸着基材を載置して化学蒸着を行なうことを特徴とする化学蒸着法。

3. 発明の詳細な説明

（産業上の利用分野）

本発明は基材表面に効率的に化学蒸着を行なう為の方法に関する。

（従来技術）

従来基材表面に化学蒸着を行なう方法として，支持体は，高温では主に人造黒鉛を用い，円錐形状の頂点で点接触状態で支持する方法が使用されている。しかしこの方法では基材と支持体とが接する部分には蒸着膜（被膜）が形成されず跡が残る為，裏返すか又は支持位置をずらすかして，2回の蒸着を要する欠点があつた。

（発明の目的）

本発明は，上記欠点を解消し，支持跡の付かない化学蒸着法を提供することを目的とする。

発明者等は研究を重ねた結果，熱分解炭素又は炭化珪素で被覆した炭素繊維の網を使用することにより，蒸着される基材の炭素繊維と接する部分に跡が付かず，基材全面に化学蒸着を施こすことができることを見出し本発明を完成するに至つた。

（発明の構成）

本発明は，熱分解炭素又は炭化珪素を被覆した炭素繊維の網からなる支持体の上に被蒸着基材を載置して化学蒸着を行なうことを特徴とする化学蒸着法に関する。

本発明に用いる炭素繊維は，原料，熱処理温度に関係なくあらゆるものが使用可能である。網への熱分解炭素又は炭化珪素の被覆は公知の蒸着法による。熱分解炭素又は炭化珪素の被覆する原料に制限はないが，熱分解炭素の場合はメタン，プロパン等の~~ブタン系~~脂肪族炭化水素，ベンゼン，トルエン等の芳香族炭化水素，ジクロロエチレン，トリクロロエタン等の有機塩素化合物など，炭化珪素

の場合は例えばメチルクロロシラン，珪素源として四塩化珪素，トリクロロシラン等，炭素源として四塩化炭素，トルエン等が好ましい。蒸着温度は原料の種類によるが通常６００～２２００℃である。基材に化学蒸着を行なうには，支持体の上に基材を載置し公知の方法により行なう。蒸着する際の圧力はできるだけ低い方がよいが，同時に蒸着速度も低下するので数mmHg付近にすれば支持体と接する部分にも十分に蒸着できて好ましい。

（作用）

上記熱分解炭素又は炭化珪素を被覆した支持体の上に被蒸着基材を載置して化学蒸着を行なう場合，支持体の炭素繊維と基材とは線接触となり，しかも支持体の表面の微細な凹凸により支持体と基材との間にも反応ガスが侵入して基材全面に蒸着被膜が形成され，支持体には熱分解炭素又は炭化珪素が被覆されているから化学蒸着後の基材と支持体は固着せず容易にはがれる。

（実施例）

以下に実施例を説明する。

実施例１

高周波誘導加熱炉の水冷式石英管（内径１００㎜）の中に，第１図(a)の平面図及び(b)の側面図に示すように炭素繊維の網（東レ株式会社製，商品名トレカＴ３００）２を外径５０㎜，内径４０㎜，厚さ５㎜の二つの人造黒鉛の枠３で挟んで固定した支持体１を入れ，１mmHgの減圧状態で１８００℃に加熱し，プロパン２０容量％を含む窒素ガスを大気圧の流量で毎分３ℓずつ１時間流し，支持体に熱分解炭素を蒸着被覆した。熱分解炭素の膜厚は４０μmであつた。

実施例２

第１図に示す支持体１を実施例１と同様にして１mmHgの減圧状態で１４００℃に加熱し，四塩化珪素9×10^{-4}モル／分，トルエン3×10^{-4}モル／分及び水素ガスを大気圧の流量で３ℓ／分の割合で１時間流し，支持体に炭化珪素を蒸着被覆した。炭化珪素の膜厚は６０μmであつた。

比較例

人造黒鉛材を加工し第２図(a)の平面図，(b)の側面図に示すような高さ１０㎜の円錐状突起５を５０㎜φ×１０ｈの円板部６の中心から１２０°ごとに等距離の位置に設けた支持体４を作成した。

上記実施例及び比較例の支持体の上に，外径３０㎜及び厚さ５㎜の人造黒鉛円盤の基材を載置して，実施例１及び実施例２と同じ条件で熱分解炭素及び炭化珪素を蒸着する実験を行なつた結果を第１表に示した。

第１表

	実施例１	実施例２	比較例	
支持体の種類	炭素繊維	炭素繊維	人造黒鉛	
支持体被膜	熱分解炭素	炭化珪素	なし	
基材への蒸着被膜	熱分解炭素	炭化珪素	熱分解炭素	炭化ケイ素
基材上面の被膜厚さ	４０μm	６０μm	４０μm	６０μm
支持部分の被膜厚さ	３５μm	５０μm	０（固着）	０（固着）

第1表から明らかなように，比較例の支持体を用いた場合は基材の支持部分に蒸着されず基材と支持体が固着したのに対し，実施例の場合は支持部分の被膜厚さは他の部分より若干薄い程度であり，基材と支持体が固着することはなかつた。

上記実験では1回の蒸着で基材1枚であつたが小さい基材ならば，又は大きい加熱炉及び支持体を使えば多量の基材の蒸着処理が可能である。

（発明の効果）

本発明によれば，基材が支持体に固着することがなく，1回の作業で基材全面に蒸着被膜を形成することができ，効率的な化学蒸着が可能となる。

4. 図面の簡単な説明

第1図は，本発明の実施例になる支持体で(a)は平面図，(b)は側面図，第2図は比較例の支持体で(a)は平面図，(b)は側面図である。

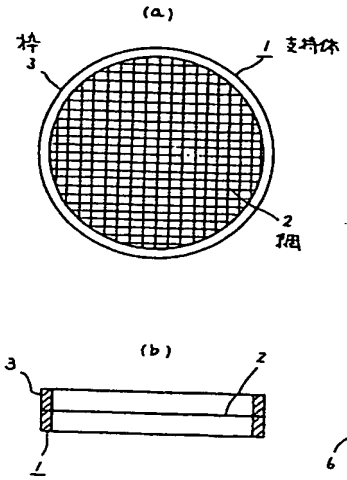
符号の説明

1…支持体　　2…網

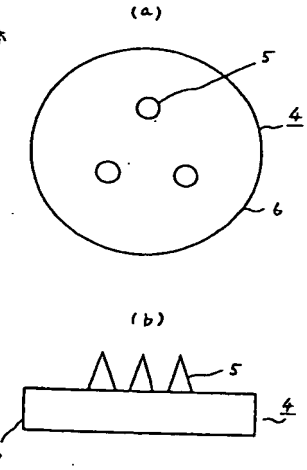
3…枠　　4…支持体

5…円錐状突起　　6…円板部

第1図



第2図



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Specification

1. Title of the Invention

CHEMICAL EVAPORATION METHOD

2. Claims

1. A chemical evaporation method characterized in that a substrate that is to be subjected to evaporation is placed on a support made of a mesh of carbon fibers covered with pyrolyzed carbon or silicon carbide, after which chemical evaporation is performed.

3. Detailed Description of the Invention

(Field of Industrial Utilization)

The present invention relates to a method for performing chemical evaporation in an efficient manner on the surface of a substrate.

(Prior Art)

A method in which mainly artificial graphite is used as a support at high temperatures, and support is provided in a state of point contact at the apex of a circular conical shape, has been used in the past as a method for performing chemical evaporation. However, such a method suffers from the following drawback: namely, no evaporation film (coating) is formed in the areas where the substrate and support contact each other, so that marks remain; accordingly, evaporation must be performed a second time after the substrate is turned upside down or after the supporting position is shifted.

(Object of the Invention)

It is an object of the present invention to eliminate the abovementioned drawback, and to provide a chemical evaporation method that leaves no support marks.

As a result of extensive research, the present inventors discovered that by

using a mesh made of carbon fibers covered with pyrolyzed carbon or silicon carbide [as a support], it is possible to perform chemical evaporation over the entire surface of a substrate without leaving marks in the areas where the substrate on which evaporation is performed contacts the carbon fibers. This discovery led to the perfection of the present invention.

(Constitution of the Invention)

The present invention relates to a chemical evaporation method which is characterized in that a substrate that is to be subjected to evaporation is placed on a support consisting of a mesh of carbon fibers covered with pyrolyzed carbon or silicon carbide, after which chemical evaporation is performed.

All types of carbon fibers, regardless of the raw material or heat treatment temperature used, may be used as the carbon fibers of the present invention. The coating of the mesh with pyrolyzed carbon or silicon carbide is accomplished by a universally known evaporation process. There are no restrictions on the raw materials used for this coating with pyrolyzed carbon or silicon carbide. However, in the case of pyrolyzed carbon, it is desirable to use an aliphatic hydrocarbon such as methane, propane or the like, an aromatic hydrocarbon such as benzene toluene or the like, or an organo-chlorine compound such as dichloroethylene, trichloroethane or the like, [as the raw material], and in the case of silicon carbide, it is desirable to use (for example) methylchlorosilane, or to use silicon tetrachloride, trichlorosilane or the like as a silicon source, and to use carbon tetrachloride, toluene or the like as a carbon source. The evaporation temperature depends on the types of raw materials used, but is ordinarily 600 to 2200°C. In order to perform chemical evaporation, the substrate is placed on the support, and chemical evaporation is performed by a universally known method It is desirable that the pressure during evaporation be as low as possible. At the

same time, however, the evaporation rate drops; accordingly, if the pressure is set in the vicinity of several mmHg, evaporation can also be sufficiently performed in the areas contacting the support, so that such a pressure is desirable.

(Effect)

In cases where chemical evaporation is performed with the substrate that is being subjected to evaporation placed on the abovementioned support covered with pyrolyzed carbon or silicon carbide, the carbon fibers of the support and the substrate are in linear contact, and the reaction gas also enters the spaces between the support and the substrate as a result of the fine indentations and projections on the surface of the support, so that a evaporation film is formed over the entire surface of the substrate. Furthermore, since the support is covered with pyrolyzed carbon or silicon carbide, the substrate following chemical evaporation and the support do not adhere to each other, and can easily be separated.

(Examples)

Examples will be described below.

Example 1

A support 1 in which a carbon fiber mesh (manufactured by Toray K.K., commercial name Torayka T300) 2 was clamped and fastened between two artificial graphite frames 3 with an external diameter of 50 mm, an internal diameter of 40 mm and a thickness of 5 mm as shown in a plan view in Fig. 1 (a) and a side view in Fig. 1 (b) was placed inside a heating furnace (internal diameter 100 mm), and was heated to 1800°C under a reduced pressure of 1 mmHg. Nitrogen gas containing 20 vol % propane was caused to flow through for 1 hour at the rate of 3 liters/min (flow rate at atmospheric pressure), so that the support was covered with pyrolyzed carbon by evaporation. The thickness of the pyrolyzed

carbon film was 40 μm.

Example 2

The support 1 shown in Fig. 1 was heated to 1400°C under a reduced pressure of 1 mmHg in the same manner as in Example 1; then, for 1 hour, carbon tetrachloride was caused to flow through at the rate of 9×10^{-4} mol/min, toluene was caused to flow through at the rate of 3×10^{-4} mol/min, and hydrogen gas was caused to flow through at the rate of 3 liters/min (flow rate at atmospheric pressure), so that the support was coated with silicon carbide by evaporation. The thickness of the silicon carbide film was 60 μm.

Comparative Example

A support 4 was prepared by working an artificial graphite material so that circular conical projections 5 with a height of 10 mm (as shown in a plan view in Fig. 2 (a) and a side view in Fig. 2 (b)) were formed at intervals of 120° in positions equidistant from the center of a disk part with a diameter of 50 mm and a thickness of 10 mm.

An experiment was performed in which substrates each consisting of a disk of artificial graphite with an external diameter of 30 mm and a thickness of 5 mm were placed on the supports of the abovementioned examples and comparative example, and were coated with pyrolyzed carbon and silicon carbide under the same conditions as in Example 1 and Example 2. The results obtained are shown in Table 1.

Table 1

Type of support	Example 1	Example 2	Comparative Example	
Material of support	Carbon fibers	Carbon fibers	Artificial graphite	
Support coating film	Pyrolyzed carbon	Silicon carbide	None	
Coating film formed on substrate by evaporation	Pyrolyzed carbon	Silicon carbide	Pyrolyzed carbon	Silicon carbide
Thickness of coating film on upper surface of substrate	40 μm	60 μm	40 μm	60 μm
Thickness of coating film in supported areas	35 μm	50 μm	0 (adhesion)	0 (adhesion)

As is seen from Table 1, there was no evaporation in the supported areas [of the substrate], and the substrate and support adhered to each other, in cases where the support of the Comparative Example was used. On the other hand, in the case of the Examples, the coating film thickness in the supported areas was merely slightly thinner than in other areas, and there was no adhesion of the substrate and support to each other.

In the above experiments, a single substrate was used in a single evaporation operation. However, if the substrates are small, or if a large heating furnace and support are used, large quantities of substrates can be subjected to a evaporation treatment [at one time].

(Merits of the Invention)

The present invention makes it possible to form a evaporation coating film over the entire surface of a substrate in a single operation without any

adhesion of the substrate to the support, so that efficient chemical evaporation is possible.

4. Brief Description of the Drawings

Fig. 1 shows the support used in the examples of the present invention; Fig. 1 (a) is a plan view, and Fig. 1 (b) is a side view. Fig. 2 shows the support used in a comparative example; Fig. 2 (a) is a plan view, and Fig. 2 (b) is a side view.

Explanation of Reference Numerals

1 Support

2 Mesh

3 Frames

4 Support

5 Circular conical projections

6 Disk part

FIG. 1

1 SUPPORT

2 MESH

3 FRAMES